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A designed experiment in a continuous process

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ABSTRACT

This paper discusses the design and analysis of an experiment performed in a continuous process (CP). A full factorial design with replicates is used to test three types of pellets on two levels of a process variable in an experimental blast furnace process. Issues and considerations concerning the experimental design and analysis are discussed. For example, an adaptive experimental design is used. We propose a multivariate approach to the analysis of the experiment, in form of principal component analysis combined with analysis of variance. The factorial design in CPs is found to have a promising potential. However, CPs also demand special considerations when planning, performing and analyzing experiments, and therefore further development of experimental strategies and connected methods of analysis for CPs is needed.

Keywords: Experimental design; factorial experiments; multivariate data analysis; principal component analysis; blast furnace experiments; split-plot design.

INTRODUCTION

The need to experiment frequently arises when trying to increase process knowledge in industry. The field of Design of Experiments (DoE) deals with methods for efficient experimentation, i.e. deriving required information about, e.g. a process, at the least expenditure of resources (Barker, 1994). Factorial designs are important tools in DoE and are exhaustively treated in literature; see e.g. Box *et al.* (2005) and Montgomery (2005). Discontinuous processes, i.e. processes where parts or batches are produced, dominate applications of DoE in practice as well as in literature. Continuous processes (CPs), which together with batch processes are frequent in process industry, use nondiscrete materials (Dennis & Meredith, 2000). In CPs the product gradually and with minimal interruptions passes through a series of different operations and the product exhibits characteristics such as liquids, powders, slurries, and pellets (Fransoo & Rutten, 1994). DoE applications in CPs have attracted considerably less attention and Vanhatalo & Bergquist (2007) argue that CPs warrant special consideration due to their characteristics. Examples of design and analysis of experiments in CPs are scarce in literature. Yet, as we will show in this article, the call for experiments in CPs does appear in industry. Consequently, there is a need for further research on experimental design and connected methods of analysis for CPs.

The purpose of this paper is thus to describe experiences from using a factorial design when doing experiments in a CP. More specifically, we discuss experimental design issues and connected methods of analysis. Furthermore, we address special considerations and deliberations that were needed during the experimental effort.

METHOD

Branches of industry, where continuous processes can be found, are the pulp and paper industries, chemical industries, parts of medical and food industries, as well as parts of mining and steel industries, and suitable cases to study are likely to be found in any of these. In this work, an Experimental Blast Furnace (EBF) operation was selected as a case, because the research engineers at the EBF plant (EBF engineers) were interested in developing their experimental designs in general and testing factorial experiments in particular.

The experimental effort at the EBF plant can be partitioned into three phases: pre-experimental planning; performing the experiment; and doing the analysis. A team consisting of EBF engineers and scholars in quality technology and statistics collaborated during all these phases. Also, observations and interviews were used as complementary data collection methods. During the pre-experimental planning phase a checklist, developed from recommendations by Coleman & Montgomery (1993), was used to structure the experimental planning activities; see Vanhatalo & Bergquist (2007).

The Experimental Blast Furnace (EBF)

The EBF was inaugurated in 1997 by Luossavaara-Kiirunavaara AB (LKAB), a leading Swedish producer of highly developed iron ore products (pellets in particular). The EBF is a pilot scale blast furnace, specifically designed for experimental use and intended mainly for product development but also to improve knowledge about LKAB's customers' process - the blast furnace process. The blast furnace can be characterized as a high temperature counter current reactor for reduction and smelting of iron ore into hot metal (Geerdes *et al.*, 2000). Coke and coal are used to reduce iron oxide, normally in form of sinter and/or pellets, into liquid iron. The production capacity is approximately thirty-five tons of hot metal per day (compared to up to 10,000 tons/day for the largest full scale furnaces).

The pilot scale enables a realistic, controlled and safe way to conduct experiments and it is possible to create reactions and progress that can be expected of full scale blast furnaces. Also, the response time in the EBF is much shorter than in commercial furnaces. The experimental costs and risks associated with performing experiments are great even in this scale but they are substantially lower than in full scale operation. An outline of the EBF and examples of measurement possibilities are presented in Figure 1.

Most experiments performed in the EBF include response variables connected to the output from the process, such as chemical composition of iron and slag, as well as process response variables related to energy efficiency and stability of the process itself, e.g. gas utilization. Both product quality and efficiency and stability of the process are important responses to the customer.

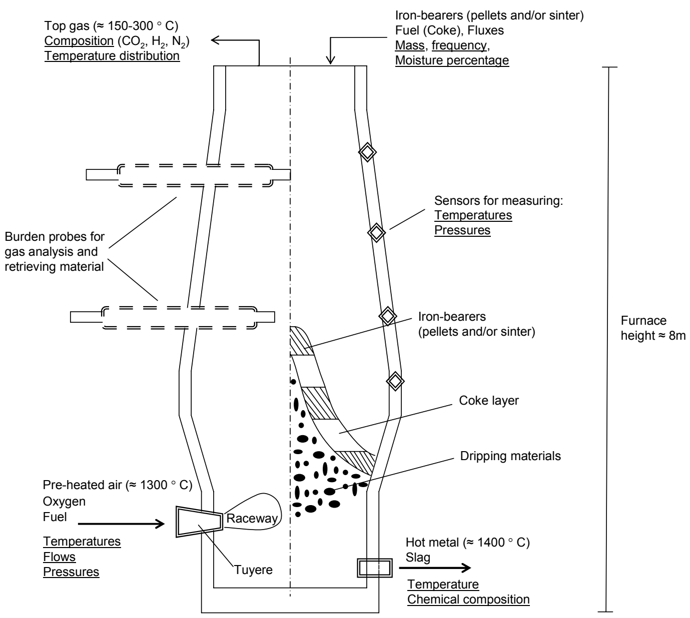

Figure 1. Outline of the EBF process. Examples of possible measurements are underlined.

BACKGROUND AND PRE-EXPERIMENTAL PLANNING

The main aim of the experiment was to investigate if three different types of pellets (here called A, B, and C) significantly differ with respect to properties and performance in the EBF process on two levels of a process variable. The chemical composition of the A and B type pellets were changed, compared to the standard product C, in order to try to induce a possible beneficial effect on their performance in the blast furnace.

In addition, "blast volume" was also considered as an experimental factor. In metaphor, the blast volume could be compared to the "throttle" of the process and increasing the blast volume would result in a higher production rate, as well as shorter dwell time for the pellets in the shaft, and thus a shorter time for the reduction work. Beforehand, a hypothesis was that increasing blast volume would "stress" the pellets to a higher extent, and thereby possible differences in the material strength and performance could be highlighted in process performance. Two levels of blast volume were chosen to be tested (1600 and 1800 Nm^3/h). The choice of factors and levels was preceded by extensive discussion, which is not elaborated here. The same goes for factors to be held constant as well as disturbance factors and connected strategies to offset them.

We had two experimental factors; one at three levels and the other at two levels. The basic 3x2 factorial design requires six runs for each full replicate. The time available in the EBF was limited to fifteen days in total and replication of runs was considered crucial. Hence, the next question to consider was how many replications were possible. This question was intimately connected to the *dynamic* characteristic of the EBF process, i.e. process inertia; see Black-Nembhard & Valverde-Ventura (2003). Twenty-four hours for each run was judged to provide enough replicates but instead there was a concern that this time could be too short in relation to the process' inherent inertia, i.e. the time needed for the responses of interest to react to the changes of the experimental factors. Thirty-six hours per run was also considered but was rejected as too few replications would be possible, in the light of the probability for one or a few runs to be excluded due to process disturbances. Therefore, investigations were made with purpose to settle the time needed for change-overs of material to manifest themselves in response variables of interest. This was made arguing that the responses in iron and slag would be the "slowest" to react among all responses, and it was concluded that about six to eight hours was needed. Consequently, using twenty-four hours for each run would leave about sixteen hours for analysis after excluding the first eight hours in each run from the data. However, this made the design sensitive to disturbances as there was a risk of not being able to isolate any reliable analysis period in data for the run if disturbances occurred. Therefore, it was decided to use what we choose to call an "adaptive design", i.e. the planned time for each run was twenty-four hours but could be extended if a disturbance occurred, e.g. process equipment failures. By using the adaptive design the prospect was to at least manage a full replication of the basic 3x2 design, i.e. at least twelve individual runs.

An important experimental complication is that process control during experimentation is unavoidable in the EBF process. Without controlling fuel ratio during experiments in the EBF, there is a risk for the melt to freeze or overheat, which can jeopardize the experimental campaign, the plant as well as personal safety. The melt temperature control of the EBF is further complicated since it is manual, requiring human deliberations at certain processing states, and the control includes a large but often unknown time lag. Since only twenty-four hours were planned for each run in the design it was therefore important to develop a strategy to maintain a good thermal state in the process when switching between runs, and to control the process by several minor adjustments in fuel rather than less and bigger adjustments.

The run order was not completely randomized, since all three types of pellets could not be acquired in good time before the experiment. Also, the blast volume was set to be on the same level for a couple of runs at a time in order to facilitate process control, especially during the six first runs of the design. It was also decided to start the experiment by running the process at 1800 Nm^3/h in blast volume due to the remaining uncertainty about the appropriateness of this choice of high level for the blast volume factor. If problems were to occur then the corresponding run(s) could be excluded from the design, the levels of the blast volume reset, and the experiment continued only loosing a smaller part of the valuable experimental time in the EBF. With exception for the first run, the type of pellets in each run was randomly assigned with the restriction that each run would imply a change of pellet type. The experimental design is summarized in Table I.

Table I. The run order of the twelve performed runs in the 3x2 factorial experimental design.

Run number	**Pellets type**	**Blast volume** [Nm^3/h]	**Hours available for analysis**	**Run number**	**Pellets type**	**Blast volume** [Nm^3/h]	**Hours available for analysis**
1	C	1800	24	7	B	1800	17
2	A	1800	17	8	C	1800	22
3	B	1800	17	9	A	1600	17
4	C	1600	17	10	C	1600	10
5	B	1600	17	11	A	1800	19
6	A	1600	17	12	B	1600	19

PERFORMING THE EXPERIMENT

The adaptive design was a useful strategy to cope with the disturbances that did occur. In runs 1 and 8 (see Table I), problems with malfunctioning process equipment affected the process to such an extent that the decision was made to prolong these runs for a couple of hours respectively. During run 10, there was a snowstorm during which a material conveyor belt broke and for a couple of hours no pellets could be transported into the EBF plant. This did of course affect the process, but since there was a shortage of type C pellets the run could not be prolonged. Hence, run 10 provides the smallest number of hours of normal operation for analysis.

In a best case scenario fifteen individual runs would have been possible, but due to disturbances during previous runs these could not be performed. All in all 12 runs were conducted, i.e. two replicates of the 3x2 factorial design.

DOING THE ANALYSIS

We start by presenting an overview of the steps in the analysis process in Figure 2.

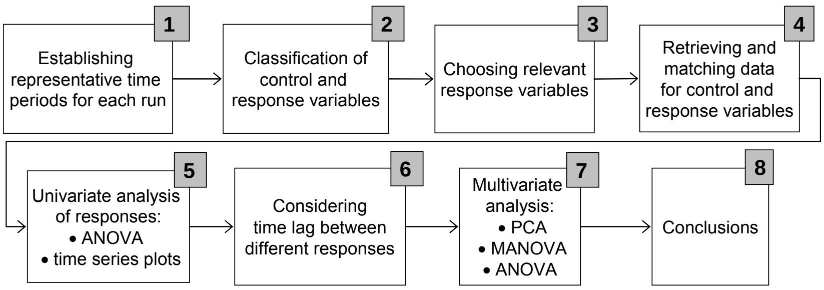

Figure 2. The main activities of the experimental analysis process.

At the outset, time periods representing normal process operation, where no apparent disturbances were present, needed to be established. In doing this about eight hours of process data were excluded in connection to each change-over between experimental runs. Logbooks and visual inspection of time series plots over important variables were used in this step. Next, response variables of interest were classified and selected from the many possible responses that are logged continuously in the process. In doing this we had to consider that several of the response variables were in fact calculated from the same sensors in the furnace or from other response variables. We therefore tried to avoid including more than one variable carrying on exactly the same information. However, the remaining responses are still correlated to different degrees.

An arduous work to retrieve and match data from the process monitoring system was the next step and this included reworking of data matrices to get the same resolution of the data.

Univariate analysis of responses was first considered as a possible way to analyze the data, i.e. generating separate ANOVA tables for all the responses of interest as well as studying univariate time series plots. However, after completion it did not provide the fundamental overview of the experimental results. Moreover, by making so many separate ANOVAs and using an individual significance level of 5 % there was an increased risk to find effects that were not real effects.

Before progressing to multivariate analysis, time lag between different responses was considered. The time-delay (inertia) differs between different response variables, e.g. changes in carbon injection feed rate would influence the composition of the furnace exhaust gas immediately, while it may take hours until such change would affect the liquid metal composition. Based on the EBF engineers' experience we chose to use a time lag of four hours between process response variables and output responses (iron and slag).

Next, principal component analysis (PCA) was conducted in order to summarize the many and correlated responses in the process and try to derive latent variables that could be used to interpret the "strongest" signals in response data; a description of PCA can be found in, e.g. Johnson & Wichern (2002).

The PCA was conducted on a data matrix consisting of 213 observations (hours of operation) and 52 response variables (mean-centered and scaled to unit variance); see Figure 3 for a summary of the data matrix.

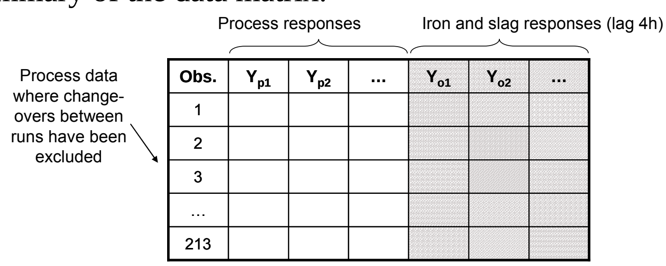

Figure 3. A summary of the data matrix (213 observations and 52 variables) used in PCA. The response variables are divided into "process responses" (Y_{px}) and lagged "output responses" (Y_{ox}).

The software SIMCA-P+ 11.0 (Umetrics AB) was used to conduct the PCA. A model with seven principal components (PCs) explained 75.2 % of the total variation in the data; see Table II.

Table II. Explained variance (of the response space) and eigenvalue for each principal component.

Principal component number:	Eigen-value	Explained variance	Cumulative explained variance	Principal component number:	Eigen-value	Explained variance	Cumulative explained variance
1	13.20	25.3 %	25.3 %	5	3.60	6.9 %	66.4 %
2	9.56	18.4 %	43.7 %	6	2.49	4.8 %	71.1 %
3	4.23	8.1 %	51.8 %	7	2.10	4.0 %	75.2 %
4	3.96	7.6 %	59.4 %				

Principal component (PC) score plots were visually examined in order to investigate if and how the experimental factors affected the process. Visual effects of the experimental factors could be traced in the first four components. This way of working, labelling the score plots according to the levels of the factors, is exemplified in Figure 4a-b. Note the clear separation of the two levels of blast volume in Figure 4a as well as the generally higher values on PC4 for the type "C" pellets in Figure 4b.

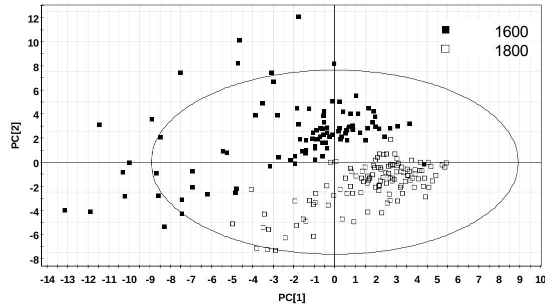

Figure 4a. Scatter plot of the PC1-PC2 scores labeled according to the level of blast volume.

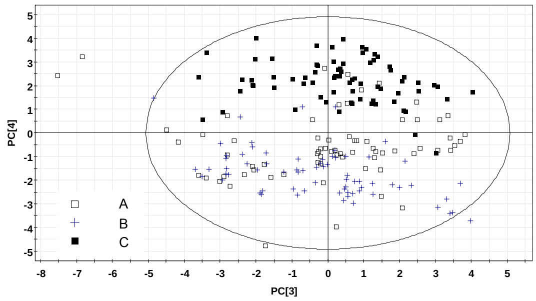

Figure 4b. Scatter plot of the PC3-PC4 scores, labeled according to the type of pellets.

By visual inspection of the score plots it became evident that the blast volume factor seemed to affect the scores of the first three principal components, i.e. it seemed to have the largest effect on the processing state; see e.g. in Figure 4a. The type of pellets did not seem to have a substantial effect, but a difference in the level of PC4 was found (see Figure 4b). PC loadings were then used to interpret what the dimensions of the PCs mainly describe. PC1 appeared to describe a dimension affected by the general thermal state in the process. PC2 can be viewed as an indicator of gas distribution in the furnace shaft. PC3 describes the gas utilization (efficiency) in the process. PC4 was interpreted as a chemical dimension of the iron. No sensible interpretation could be made of the subsequent PCs.

The effect of the experimental factors could be visually detected in a subspace of the first four PCs, but was there really a significant difference in the PC scores depending on the experimental factors? To answer this question the PC scores for PC1-PC5 were averaged for each of the twelve runs of the experiment. The scores were averaged since there was a high degree of autocorrelation in the data. Also, multivariate analysis of variance (MANOVA) and ANOVA assume independently and normally distributed observations, which are reasonably achieved by calculating averages.

Next, a MANOVA was performed, using Statgraphics Centurion XV software, on the averages of the PC scores. At 5 % significance level the MANOVA showed significant main effects of the experimental factors, with e.g. Wilks' Lambda p-values of 0.041 and 0.013 for pellets and blast volume respectively. The interaction effect was not significant. Thereafter, separate ANOVAs for the five PCs were made on the average of each PC in the twelve runs; see Table III.

Table III. P-values from the ANOVAs for the averages of the PC scores for each of the twelve runs. Values in brackets are from the split-plot method of analysis. (*<0.10; ** <0.05; and ***<0.01)

Effects	PC[1]	PC[2]	PC[3]	PC[4]	PC[5]
Pellets (P)	.916 (.900)	.464 (.284)	.655 (.443)	.0003*** (.0012***)	.731 (.607)
Blast volume (BV)	.051* (.171)	.008*** (.110)	.043** (.219)	.475 (.597)	.599 (.722)
Interaction (PxBV)	.904 (.887)	.969 (.939)	.861 (.731)	.177 (.152)	.703 (.573)

From Table III we can see that the blast volume has a significant effect on the average of the PC scores for the first three PCs. Also, there is a significant pellets effect on the fourth PC. Since the first four PCs together account for almost 60 % of the variation in process data, with diminishing explanatory ability for each successive PC, we conclude that the blast volume factor has the largest effect on the processing state. The pellet types do not seem to have a pronounced effect on the processing state. However, this analysis assumes that all factors are subject to the same experimental error. This is not the case here where the randomization of the blast volume factor has been restricted. Therefore, a split-plot analysis has been performed (see Table III) where the blast volume has been treated as the whole plot factor; see Box *et al.* (2005). Even though the randomization in the design has not been restricted as much as in a split-plot design this analysis provides an idea about relevance of the p-values in Table III. We conclude that the significance of the blast volume factor on the first three PCs is probably overestimated when assuming the same experimental error for all factors. Still, we argue that it is reasonable to assume that the blast volume significantly affects the first three PCs and that the true p-values lie somewhere between the two extremes given in Table III. That the blast volume would affect the process significantly was expected. Nonetheless, since this effect was distinctly displayed in the results, it strengthens the acceptance of the overall results and the method of analysis. The interaction effect between the types of pellets and the blast volume that was discussed beforehand has not been detected in data.

To explain what causes the effects on the PCs, PC loading plots were studied. For example, the pellets effect on PC4 is mainly due to difference in the chemical composition of the produced iron, which could be expected since the chemical composition, e.g. phosphorous content of the pellets, is different. There is, however, no clear evidence of any other difference in blast furnace performance (efficiency and stability) for the three pellets that were tested.

CONCLUSIONS AND DISCUSSION

Conducting a factorial type experiment in this continuous process setting was not straightforward. Examples of the considerations that needed to be made are: the length of the individual runs in the design was affected by the dynamic characteristic of the process; an adaptive design was needed in order to try to offset the effects of disturbances during the experiment; and complete randomization of the run order needed to be abandoned. The adaptive design (being able to prolong runs if disturbances occurred) was useful when trying to cope with the disturbances that did occur. Without the adaptive design, runs 1, 8, and 10 would have been excluded from the experiment, which would have been costly (valuable experimental time would have been lost). However, this means that we have a different number of hours for analysis for the different runs in the experiment. Run 10 has the smallest number of hours for analysis. Since the hours of operation have been averaged during analysis this will affect the spread of the average for run 10. However, no problem has been detected during residual analysis connected to the ANOVA.

Due to the many and correlated response variables, a univariate approach to analysis was abandoned as it did not provide the fundamental overview of the results. Instead, we argue that in processes like the EBF, where responses are abundant, frequently logged, and correlated, a multivariate approach is beneficial. In this case we propose that PCA can be used to derive latent, uncorrelated variables that summarize the variation in the process. These can then be used as responses to test for statistical significance of the effects of the experimental factors; in this paper MANOVA and ANOVA has been applied to averages of PC scores for the runs in the experiment. To use Partial Least Squares (PLS) to analyze the data has been considered, but due to the qualitative pellets factor this technique was abandoned. The need to restrict the randomization order of the experiment to facilitate process control does however affect the analysis as all factors can not be assumed to be subject to the same experimental error. In retrospect, it would have been better to design the experiment as a regular split-plot design, with blast volume as whole-plot factor and pellets as subplot factor. In this way, fewer changes in blast volume would have been necessary. Furthermore, the statistical analysis would have been more straightforward. The design in this experiment is somewhere midway between a completely randomized design and a split-plot design.

Further, the need for process control of the fuel ratio during experimentation is a complicating matter. Even though a control strategy was developed beforehand and followed during the experiment, the manual control still continues to create ambiguity about experimental results among the experimenters. Questions like *"did a decision to add or subtract fuel at the wrong time hide important experimental results?"* can always be raised. Still, we argue that in this type of complex process setting, there is no such thing as a "perfect" experiment.

Lastly, using factorial type designs, combined with the method of analysis presented here, in this continuous blast furnace process show a promising potential. Further, as is shown in this article, CPs demand special considerations from the experimenter when planning, performing and analyzing experiments. These considerations are probably affected by the specific characteristics of the process in which the experiments are to be conducted. We therefore argue that there still is much to be done to develop general experimental strategies and connected methods of analysis for CPs.

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REFERENCES

Barker, T. B. (1994). *Quality by experimental design.* New York, Dekker.

Black-Nembhard, H. and Valverde-Ventura, R. (2003). Integrating Experimental Design and Statistical Control for Quality Improvement. *Journal of Quality Technology*, 35(4):406-423.

Box, G. E. P., Hunter, J.S. & Hunter, W.G. (2005). *Statistics for experimenters: design, discovery, and innovation, 2nd ed.* Hoboken, N.J., Wiley.

Coleman, D.E. & Montgomery, D.C. (1993). A Systematic Approach to Planning for a Designed Industrial Experiment. *Technometrics*, 35(1):1-12.

Dennis, D. & Meredith, J. (2000). An Empirical Analysis of Process Industry Transformation Systems. *Management Science*, 46(8):1085-1099.

Fransoo, J.C., & Rutten, W.G.M.M. (1994). A Typology of Production Control Situations in Process Industries. *International Journal of Operations & Production Management*, 14(12):47-57.

Geerdes, M., Toxopeus, H., van der Vliet, C. (2004). *Modern Blast Furnace Ironmaking: an introduction,* Düsseldorf, Germany, Verlag Stahleisen GmbH.

Johnson, R.A & Wichern, D.W. (2002) *Applied Multivariate Statistical Analysis, 5th ed.* Upper Saddle River, N.J., Prentice-Hall.

Montgomery, D. C. (2005). *Design and analysis of experiments,* 6th ed. Hoboken, N.J., Wiley.

Vanhatalo, E. & Bergquist, B. (2007). Special considerations when planning experiments in a continuous process. *Submitted for publication.*